REPORT DOCUMENTATION PAGE

AFRL-SR-AR-TR-02-

0432

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
31 Oct 02

3. REPORT TYPE AND DATES COVERED
FINAL REPORT - 01 MAR 01 TO 31 MAY 02

4. TITLE AND SUBTITLE
INSTRUMENTATION TO FACILITATE HIGH PRECISION MATERIALS SYNTHESIS AND ANALYSIS WITH AN EXISTING TI-SAPPHIRE FEMTOSECOND LASER

5. FUNDING NUMBERS
F49620-01-1-0223

3484/US

61103D

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AFOSR/NL
4015 Wilson Blvd. Room 713
Arlington, VA 22203-1954

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

20030115 108

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION AVAILABILITY STATEMENT

Approve for Public Release: Distribution Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*
We have designed and built a high precision computer controlled materials synthesis and analysis vacuum apparatus which will enable state-of-the-art control for both micro-machining and inspection as well as thin film synthesis when interfaced to our University's Ti-sapphire femtosecond laser. The majority of the machining of this apparatus had to be performed at a company who has had difficulties with meeting schedules due to probable downsizing issues. This has left our project seriously behind schedule and thus only now could the final report be written and issued. The apparatus consists of a high precision gonimeter on which the laser target is held on to a motorized x-y stage. The target is able to rotate precisely to one of two quartz windows through which two ultra-high accuracy triangulating laser sensors are used to measure surface details to sub-micron precision regardless of the optical condition of the surface (diffuse or specular). The ability to precisely raster the target during ablation has dual use. In the case of synthesizing thin films, the rastering prevents cone formation in the target and thus improves film quality. When ablation is used for removal of layera from a sample such as paint or conversion coating, in preparation of further surface analysis, the rastering facilitates making a suitable window through the layer. Additionally, duplex-layered electrodes may be formed by combining synthesis and micro-machining so as to expose both layers during electrochemical analysis. The apparatus has been designed by us to meet the need for such a system at reasonable cost and because no such system to our knowledge was available on the market.

14. SUBJECT TERMS

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASS	UNCLASS	UNCLASS	

Final Report F49620-01-1-0223

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Instrumentation to Facilitate High Precision Materials Synthesis and Analysis With an Existing Ti-Sapphire Femtosecond Laser

Abstract

We have designed and built a high precision computer controlled materials synthesis and analysis vacuum apparatus which will enable state-of-the-art control for both micro-machining and inspection as well as thin film synthesis when interfaced to our University's Ti-sapphire femtosecond laser. The majority of the machining of this apparatus had to be performed at a company who has had difficulties with meeting schedules due to probable downsizing issues. This has left our project seriously behind schedule and thus only now could the final report be written and issued. The apparatus consists of a high precision gonimeter on which the laser target is held on to a motorized x-y stage. The target is able to rotate precisely to one of two quartz windows through which two ultra-high accuracy triangulating laser sensors are used to measure surface details to sub-micron precision regardless of the optical condition of the surface (diffuse or specular). The ability to precisely raster the target during ablation has dual use. In the case of synthesizing thin films, the rastering prevents cone formation in the target and thus improves film quality. When ablation is used for removal of layers from a sample such as paint or conversion coating, in preparation of further surface analysis, the rastering facilitates making a suitable window through the layer. Additionally, duplex-layered electrodes may be formed by combining synthesis and micro-machining so as to expose both layers during electrochemical analysis. The apparatus has been designed by us to meet the need for such a system at reasonable cost and because no such system to our knowledge was available on the market.

Description and Useage of Acquired Equipment

We have built an UHV vacuum chamber (see Figure 1-3, attached) containing a high precision sample positioning and inspection system, along with computer code written in C+, interfaceable to an athermal, ultra-fast, femto second Ti-Sapphire laser. Below we show in photo 4 our constructed machine. The system houses a multi-axis gymbal with x-y motorized stages for both target and substrate manipulation. The system accommodates both laser ablation of a target for film deposition onto a substrate as well as ablation from a sample (target) containing a coating or corrosion product layer that needs to be stepwise

removed in order to facilitate surface analysis by Micro-ATR IR spectroscopy, Static SIMS or small area XPS. High precision inspection is accommodated by two Laser Displacement Measuring Sensors (LDMS). The vacuum environment is essential to prevent oxidation of reactive species (such as magnesium) of importance to our studies. The UHV environment is accommodated by a 500L/sec turbo pump horizontally mounted for maximum conductance. (Magnetic bearings make this possible).

Newly developed triangulating CCD LDMS allow for accurate and timely feedback and measurement of both the ablation process as well as film deposition rates. These measurements are conducted in situ during the experiment and their accuracy is unaffected by color, surface texture and stray light. We used two of these sensors in tandem, one optimized for measuring optically diffuse materials and one optimized for optically specular materials. Since these instruments can be operated through a window, which we have mounted on a bellows, this strategy affords us the flexibility to monitor and capture very accurate measurements of the surface condition without having to remove the specimen from its vacuum environment and risk contamination. Both diffuse and specular sensor heads are capable of measuring surface profile details in the sub-micron range. Two dimensional profile maps can be captured in situ by rastering the precision XY stages of the planetary goniometer underneath the sensor heads in a grid-like pattern. The size of the beam spot of these sensors average about 40 microns in diameter. Should we require greater spatial resolution than can be currently provided by these devices, we propose using a custom assembled instrument which has recently been funded by the U.S. Army and is already installed in our facility. While this instrument shares the same core technology as those previously mentioned, it relies on a confocal laser mechanism to detect surface displacement changes. The high performance optics of this device allow for an extremely small beam size of only 2 microns in diameter. The sensor head is currently coupled to two ultra-high precision scanning XY stages and, when rastered underneath the sensor head, ablated crater morphology can be resolved with thousands of data points for an extremely precise picture of the erosion process itself. This equipment exists as a stand-alone system external to the vacuum chamber. The confocal laser head cannot be used in the same capacity as the triangulating sensor heads since its small focal length does not permit it to be used through a window.

These measurements require that the sample be manipulated linearly, radially or both in order to bring the surface of the sample to bear underneath the sensor heads. The planetary goniometer, in conjunction with its precision XY motorized stages, allows us this motion. Hence, a surface can be micro-machined, rotated underneath the sensor heads for inspection, then returned to its original machining position for further ablation. The original coordinates are stored in computer memory and the precision inherent in the goniometer mechanism itself is such that the item in question can be returned to the same location with great precision, even after being subjected to very complex and convoluted motion inside the vacuum chamber. However, in order to be able to reposition the sample after it has been removed from the chamber and either exposed to a test environment or following comprehensive surface analysis, for whatever reason, requires a more sophisticated approach. We will use a high resolution CCD camera to capture the position of the sample with reference to the camera's position, the camera itself being rigidly

mounted to the exterior of the chamber. The repositioning approach would proceed as follows: a sample material would be de-capped (its initial surface layer removed) by femto-second ablation. The sample would then be removed from the vacuum chamber and subjected to some type of weathering experiment thus altering its surface condition somewhat. The condition of the sample is then analyzed by any of the aforementioned chemical analysis techniques, whereupon it is re-introduced into the vacuum chamber for further de-capping and analysis. Upon re-introduction to the vacuum chamber, the camera is used by the operator to place the sample in the *approximate* position it occupied prior to its removal. At this point, custom software will correlate the images obtained before removal and after reinsertion to determine the final displacement of the sample. The precision stages will then be instructed to move the sample into its original location within the accuracy of 1 pixel. Ablation erosion can then recommence at the same location as before. This procedure can even be performed remotely via a network connection. This powerful combination of precision motion and digital image processing technology allows for tremendous flexibility and permits the inclusion of equipment already present in our facility since it gives us the freedom to remove the specimen from the chamber.

The vacuum chamber also affords protection against oxidation during laser ablation metal alloy film formation. Additionally the beam rastering of the target accommodated by the motorized stage prevents deep cratering and possible nonuniformity in the plume, while substrate inspection can be performed without breaking vacuum.

Current DoD Funded Programs Supported by this Proposal

Degradation Mechanisms of Multi-component Composite Coating Systems

Clive R. Clayton and Gary P.Halada Funded by ARL

The primary objective of the proposed work is to identify and quantify mechanisms of composite coatings degradation using spectroscopic techniques, including secondary ion mass spectroscopy (SIMS), laser confocal topographic measurement, digital image correlation strain vector detection, fourier transform infrared (FTIR) microspectroscopy, X-ray photoelectron spectroscopy (XPS) and UV-vis spectroscopy, and relate the resulting model of surface and interfacial chemistry to surface topographic changes (due to interfacial adhesion failure), corrosion behavior and performance, including color change. Both the planning and results of this work will be integrated with the ongoing effort at the Army Research Laboratory, Aberdeen, Maryland, which has recently been awarded a large program from the Strategic Environmental Research and Developmen Program (SERDP) to study military coatings degradation. The models and analytical test developed in this program will be integrated into military efforts in Life Cycle Analysi of CARC composite coatings. As part of this effort, the Army, Navy, Air Force an Marines are performing field-testing of CARC coatings. Representative samples will b delivered to our laboratory for analysis. In summary, the primary focus of our research i to: (1) develop suitable analytical tools, (2) develop failure mechanisms using spatiall resolved optical methods with IR and SIMS, and (3) establish failure modes of CAR coatings from field tests.

Corrosion Protective Coatings for Depleted Uranium Penetrator Systems

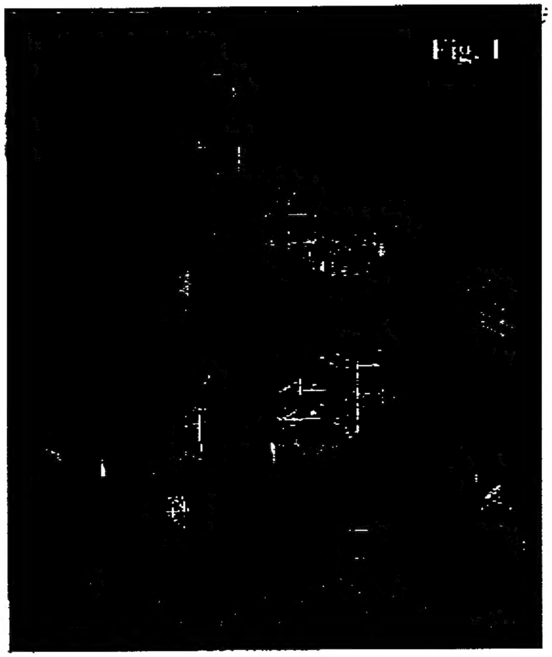
Clive R. Clayton and Gary P. Halada Funded by ARL
Depleted Uranium Penetrator sections have been prepared for electrochemical analysis in nitric acid solution. Localized corrosion has been observed to occur at the former sights of triangular uranium-titanium intermetallic compounds. We are screening a number of promising inhibitor ions which may be formed into a non-toxic conversion coating.

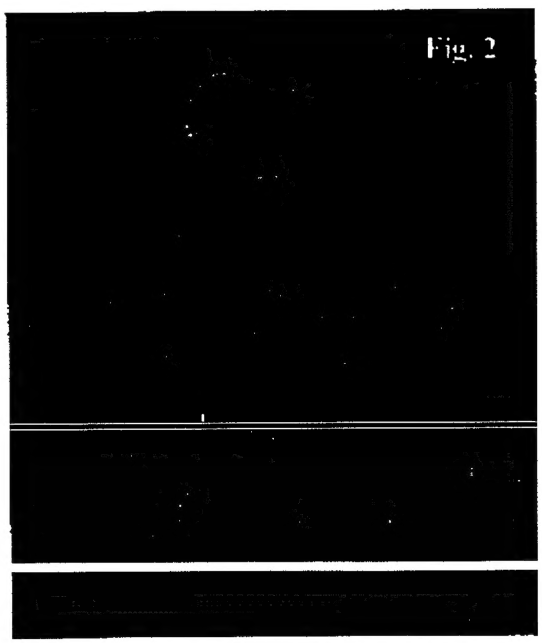
Development of New Research Capabilities In Support of Graduate and Undergraduate Training

The primary function of the system will be to extend our knowledge of mechanisms of both corrosion failure and corrosion protection and will serve to strengthen on going DoD funded graduate student research as well as to support future research and educational endeavors in the multi-disciplinary field of MEMS. The system will be the prime apparatus for undergraduate thin film design projects supervised by the P.I. (Clayton) in his course offering: ESG 339 Thin Film Processing. The instrumentation will enable new compositionally more complex thin films to be synthesized by fs laser ablation than currently may be formed in this laboratory by ion beam methods such as sputtering and ion assisted deposition. Hence, complex intermetallic compounds may be synthesized for the first time having chemical and structural homogeneity. This includes freedom from pin-holing often produced by ion beam techniques. In ESG 339 the students are taught vacuum design, surface analysis and thin film synthesis by ion beam and laser beam processing. A thermal ultra-fast laser ablation is a breakthrough method of film synthesis, machining (cutting) and analysis. The apparatus built through this DURIP award will provide cutting edge training and instruction in a new and potentially important new synthesis tool.

Costs Incurred in Equipment Manufacturing

The major part of this grant and associated institutional cost share has covered the engineering by Thermionics Northwest Inc. of our design of a complex UHV suitable goniometer, vacuum chamber and a custom eight inch diameter window which is mounted on a bellows with rack and pinion. This package cost about $60,000. The rest of the funding covered: two laser sensor heads and computer controlled x-y tables ($20,000), a 500 liter per second turbo pump with magnetic bearings ($10,000) programmable sample heater ($6000), aluminum extruded materials ($5000), two computers and monitors ($5000), 8 inch gate valve ($3000), the balance covered purchases of software, gaskets, fasteners and vacuum gauge instrumentation.





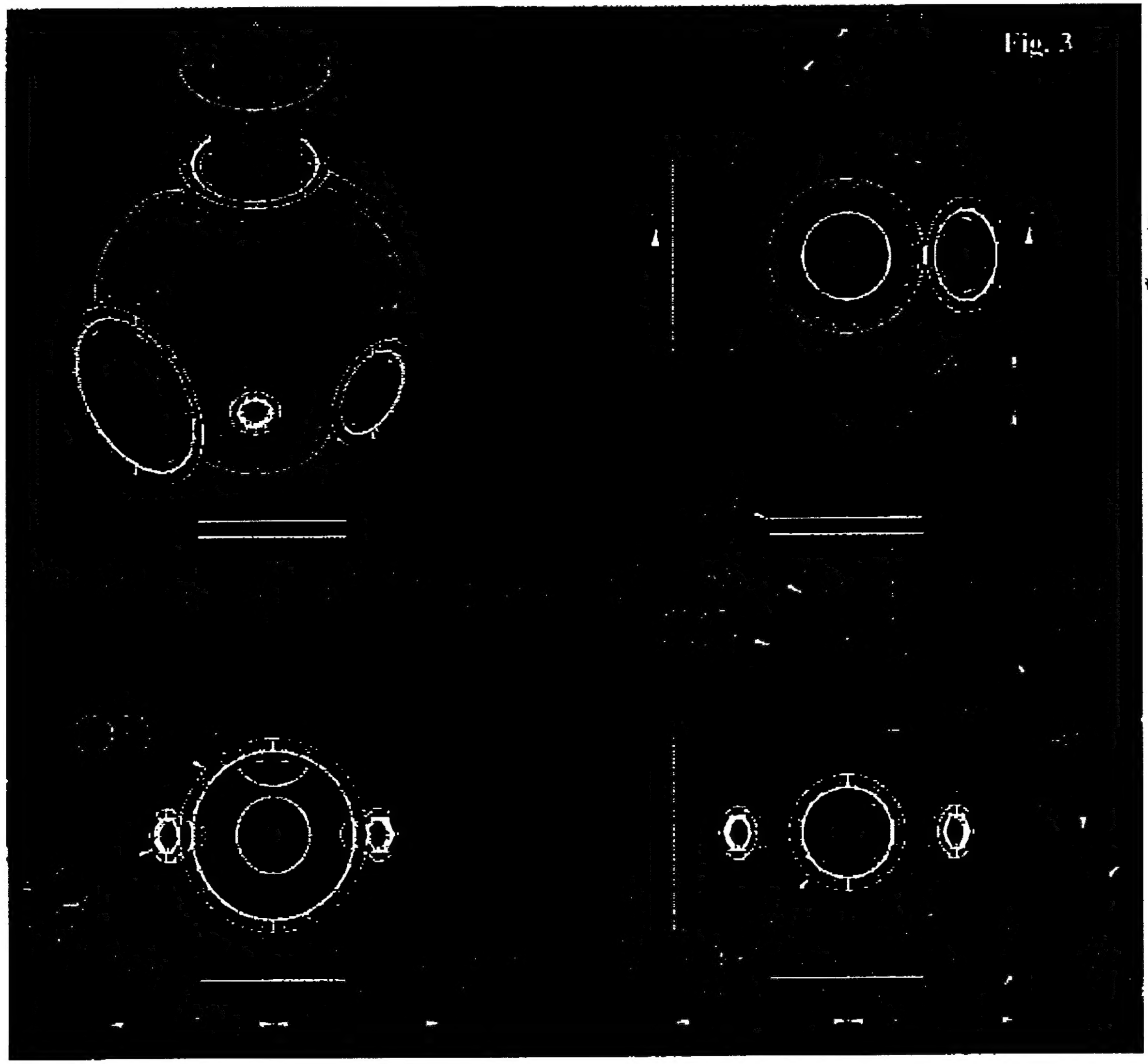


Figure 1) **3-D CAD wire frame drawing of equipment cradle (chamber not shown).**

Figure 2) **Figure 1, rendered with hidden lines removed.**

Figure 3) **Original vacuum chamber schematics.**

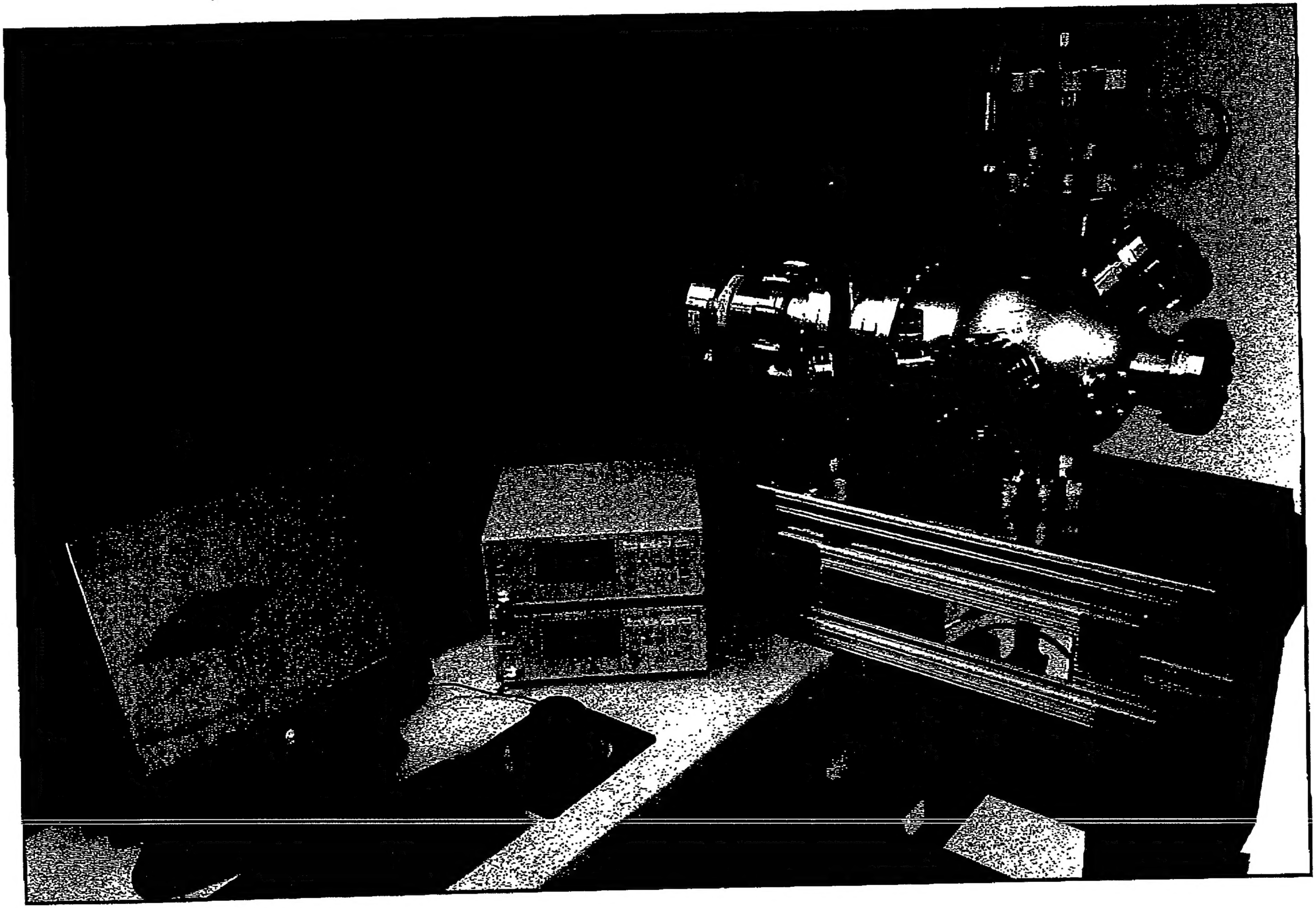


Fig. 4 – Completed system shown with computer workstation and laser sensor electronics.